NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

WARTIME REPORT

ORIGINALLY ISSUED

February 1946 as
Advance Confidential Report E5L18

OCCURRENCE OF IRON OXIDES ON CAST-IRON ENGINE

SURFACES AFTER OPERATION

By A. S. Nowick and L. O. Brockway

Aircraft Engine Research Laboratory
Cleveland, Ohio

NACA

WASHINGTON

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

ADVANCE CONFIDENTIAL REPORT

OCCURRENCE OF IRON OXIDES ON CAST-IRON ENGINE

SURFACES AFTER OPERATION

By A. S. Nowick and L. O. Brockway

SUMMARY

Surfaces of used cast-iron piston rings from various single-cylinder aircraft engine tests were examined by electron diffraction. In addition to α-iron and graphite, extra rings appeared in many of the diffraction patterns and were identified as diffraction rings of α-Fe_2O_3 and either Fe_3O_4 or γ-Fe_2O_3 (these two materials are indistinguishable by diffraction methods). In general, the oxide patterns were obtained to an appreciable extent from only the top compression ring of the piston-ring assembly; the other rings gave predominately a graphite pattern. The diffraction pattern from a new ring showed α-iron with lesser quantities of graphite but never more than traces of any other materials.

Examination of a cast-iron cylinder which had been operated for a long period of time showed that, relative to graphite and α-iron, iron oxides were present in continuously increasing amounts as the top of the piston stroke is approached. Specimens from the part of the cylinder below the region of ring travel showed no detectable quantities of oxides.

It was concluded that iron oxides are formed on cast-iron surfaces during engine operation in amounts depending upon operating variables such as temperature, pressure on the surfaces, and exposure to corrosive agents in the combustion products. In the determination of the nature of a run-in surface on cast-iron piston rings it will be necessary to consider the formation of iron oxides.

INTRODUCTION

During the operation of sliding surfaces, physical and chemical surface changes may occur A "run-in" surface is defined in reference 1 and also in this report as a surface whose properties have so altered that it yields optimum performance characteristics,

for example, a low rate of wear. Electron-diffraction techniques
are valuable in the study of the nature of run-in surfaces because
the low penetrating power of electrons often permits detection of
a substance covering the surface to a depth of only a few molecules.
Application of electron-diffraction techniques to the study of used
aircraft-engine cylinder sleeves was made by Finch, Quarrell, and
Wilman (reference 2). They obtained diffuse patterns, from which
they concluded that an amorphous Beilby layer was present. This
conclusion may not be justified in view of the work of Germer
(reference 3) and others.

An electron-diffraction examination (reference 1) of cast-iron
piston rings from single-cylinder engine tests showed that a layer
of graphite covered the surfaces of the cast-iron compression rings
as well as the steel barrel and the top chrome-plated ring of the
assembly examined. The present investigation continues the electron-
diffraction analysis of cast-iron surfaces as part of a general pro-
gram being conducted at the NACA Cleveland laboratory to determine the
chemical and physical nature of run-in surfaces. The information
obtained from this program should be of value in improving the high-
output performance of military aircraft engines by making possible
specifications of the most suitable combinations of running surfaces.

Specimens were examined from piston rings used in tests on
liquid- and air-cooled single-cylinder engines having only cast-iron
piston rings; emphasis was given to the top cast-iron compression
ring because the piston-ring assembly previously examined (reference 1)
had a chrome-plated top ring. In addition, it was desired to compare
the running surface of a cast-iron engine part that had been operated
for a long period of time with the surfaces of cast-iron aircraft
piston rings that had been run for a shorter period. A cast-iron
cylinder block that had had long service in an engine was available
and the surface of one of the cylinders was examined, although the
exact operating conditions during its life were not known. Photo-
graphs presented in this report show representative electron-
diffraction patterns obtained from the cast-iron piston rings and
comparison patterns of some of the materials involved.

APPARATUS AND TEST PROCEDURE

The cast-iron piston rings for this investigation were obtained
from ring assemblies used in four different single-cylinder tests.
The first three tests used cast-iron compression rings of rectangular
cross section (with a beveled face on the top ring and tapered faces
on the other two) operated in an SAE 4620 carburized steel cylinder
barrel in a liquid-cooled engine. The fourth test used cast-iron

compression rings (with keystone cross sections and tapered faces)
operated in a chrome-plated cylinder barrel in an air-cooled engine.
In addition to the used rings examined, specimens of a new beveled
compression ring (lapped by the manufacturer) of the type used for
the top ring of tests 1, 2, and 3 were examined for control purposes.
Operating conditions for the four tests are listed in table 1.

After the rings were removed from the engine, they were immersed
for 24 hours in a compound that loosened the "carbon" (oil combustion
products) deposited. The rings were then washed with an organic
solvent and covered with grease until just before the diffraction
exposures were made. A control test of this cleaning process was
carried out as follows: One lapped piston ring and one taken
directly from a test were broken in two and half of each ring was
put through the cleaning process. A comparison of the electron-
diffraction patterns from specimens of both the treated and untreated
halves showed that the cleaning process had not changed the nature of
the running surfaces.

A longitudinal strip one-fourth inch wide was cut from the cast-
iron cylinder and specimens about one-fourth inch in length were cut
at various measured distances from the top of the cylinder. These
distances are listed in table 2.

The comparison electron-diffraction patterns used for the
identification of unknown patterns were those of α-iron, graphite,
α-Fe$_2$O$_3$, and γ-FeOOH. The α-iron pattern was obtained from a
sample of cold-rolled steel of very low carbon content mildly abraded
with emery paper. The graphite pattern was obtained by etching cast
iron with a 2-percent nital solution (2-percent nitric acid in methyl
alcohol) for 30 seconds. A specimen of the low carbon steel, abraded,
etched with dilute aqueous nitric acid for about 30 seconds, and
finally heated until blue interference colors appeared, gave a
pattern principally of α-Fe$_2$O$_3$. A pattern principally of γ-FeOOH
was obtained from rust specimens prepared from cast-iron placed above
water at 50° C for 24 hours. It is desirable to make comparison with
diffraction patterns from known materials obtained by electron diffrac-
tion rather than by X-ray diffraction wherever possible because the
relative intensities in electron patterns may differ greatly from the
X-ray intensities as a result of the large absorption factor of the
specimens for electrons. The X-ray patterns available in the A.S.T.M.
card file are useful in checking the positions of the diffraction
rings.

The electron-diffraction camera is a commercial electron micro-
scope equipped with a diffraction adapter. The instrument was oper-
ated at 60 kilovolts and the specimen-to-plate distance was
31.0 centimeters.

The Bragg interplanar spacings d (in angstrom units A) in
the crystal were obtained from the measured values of the radii r
(in mm) of the corresponding diffraction rings. For the small
angles used in electron diffraction, d may be related to r
according to

$$d = \frac{K}{r}$$

where K is a constant that depends on the voltage and the
specimen-to-plate distance. The value of K was determined by
using the diffraction pattern from zinc oxide as a calibration pat-
tern. Over the period of time during which these experiments were
conducted, the value of K varied from 14.0 to 14.7.

The diffraction photographs presented are positive enlargements
(X2) in which some of the detail of the original negatives has been
lost.

RESULTS AND DISCUSSION

The d values for both reference and unknown materials have
been plotted in figures 1 to 4 for convenience in identification of
the unknown patterns. The method of plotting a given pattern con-
sisted in marking the d values along a horizontal axis by vertical
lines whose positions and heights show, respectively, the relative
positions and the qualitative order of intensities of the diffraction
rings in the photograph. The diffraction rings are represented by
fine lines despite the fact that the actual rings tended to be broader
as a result of beam and specimen widths. Whenever an unusually wide
ring occurred, it was plotted as a band to show that it probably
consisted of more than one ring. The scale used in plotting is
inversely proportional to the d values and corresponds to an
enlargement of the original negatives by a factor of about 8.5. The
precision of the measurements of the radii in the negatives, amounting
usually to ±0.1 millimeter, is therefore indicated by an interval of
0.85 millimeter at all positions along the scale. In the case of
very faint rings the measurements were somewhat more uncertain and
the precision is correspondingly poorer.

Identification of Iron Oxides

Electron-diffraction patterns obtained from specimens of the
various used rings listed in table 1 and from the cylinder operated
for a long period showed, in addition to α-iron and graphite, the
presence of extra diffraction rings. The intensities and the
numbers of these extra rings as compared with graphite and α-iron

rings depended upon the specimen in a manner to be discussed later
in this report. The stronger of the extra rings corresponded to
strong rings of some of the iron oxides and, accordingly, a detailed
comparison with the iron-oxide patterns was made.

Included in the data are the following comparison patterns:
electron-diffraction patterns of α-Fe_2O_3 (fig. 1(d)); graphite
and α-iron (figs. 2(d) and 2(e), 3(f) and 3(g), and 4(e) and 4(f),
respectively); and A.S.T.M. X-ray diffraction patterns of γ-FeOOH,
α-Fe_2O_3, and Fe_3O_4 (presented in figs. 1(a), 1(c), and 1(e),
respectively). Photographs of the α-iron, graphite, and α-Fe_2O_3
patterns are presented in figures 5(a), 5(b), and 5(c), respectively.
The presence of impurities is indicated by the presence of additional
rings (marked X in the corresponding comparison plots): two rings
in the α-Fe_2O_3 pattern (fig. 1) and one ring in the graphite
pattern (fig. 2). These rings are probably a result of the method
of preparation of the specimens. For example, the additional ring
in the graphite electron-diffraction pattern is probably from an
oxide of iron, formed by the slight oxidation of the iron by the
nital solution used to prepare the graphitic surface. The α-Fe_2O_3
ring at 3.68 A may not always appear in the electron-diffraction
photographs because it can be obscured by the shadow line if the
specimen extends too far into the beam or by halation of the image
of the central beam in long exposures.

A further comparison was made with the oxide formed by rusting
in humid air. Electron-diffraction patterns from various rusted
specimens were found to be very similar except for the appearance
in the diffraction patterns of dark brown rust of three strong
diffraction rings that do not appear in the patterns of the lighter,
orange rust. These three diffraction rings, which are at 2.55 A,
1.62 A, and 1.49 A, are the three strongest rings of Fe_3O_4. Fig-
ures 1(b) and 5(d) show the pattern from dark brown rust. A comparison
of this pattern with figure 1(a), the X-ray diffraction pattern of
γ-FeOOH, shows that rust is predominately γ-FeOOH with some Fe_3O_4
present in the case of this dark brown rust. This identification is
in agreement with other sources. (See reference 4.)

The electron-diffraction pattern of the specimens from piston
ring 1 of test 1 (see table 1) that gave the best oxide lines is
presented in figures 1(f) and 6(a). Comparison with the reference
patterns shows that this oxide is not γ-FeOOH but consists of
α-Fe_2O_3 and Fe_3O_4. It should be noted that γ-Fe_2O_3 and Fe_3O_4
are both cubic crystals having the same cell edge and having unit-
cell structures sufficiently alike to make them indistinguishable
by diffraction methods. For convenience, further reference in this
report to an identification as Fe_3O_4 will imply Fe_3O_4, γ-Fe_2O_3,

or both. In addition to the oxide rings, the four strongest graphite rings (marked Y in fig. 1(f)) appear, which shows the presence of a relatively small amount of graphite.

An oxide pattern from a specimen taken near the top of the cast-iron cylinder (specimen 3, table 2) is given in figure 1(g). This pattern also shows the presence of relatively little graphite and differs from the preceding pattern in the presence of three rings showing preferred orientation. Although the first of these rings coincides with the ring of α-Fe_2O_3 at 3.68 A, the other two do not agree with rings of any known oxide of iron. The three rings probably belong to the same material because their arcs subtend the same angle, which is about one-sixth of the angle of all the other rings of the pattern. The other rings (except for that at 2.30 A, marked Z in fig. 1(g)) correspond to Fe_3O_4 with traces of graphite and possibly α-Fe_2O_3.

The four unidentified lines in this pattern have occurred in several patterns and from more than one specimen. The differences in relative intensities which may occur between electron and X-ray diffraction patterns suggested that these lines, observed in electron diffraction, may have been Fe_3O_4 reflections which did not appear in the X-ray comparison pattern for Fe_3O_4. A calculation of all theoretically possible reflections, however, eliminated this possibility. A comparison of these lines with other patterns in the A.S.T.M. X-ray diffraction card file failed to identify them.

Occurrence of Oxides on Piston-Ring Surfaces

Patterns from two cast-iron piston rings of test 1 (table 1) are compared in figure 2 with patterns from the new cast-iron top compression ring, graphite, and α-iron. Except for the line at 1.31 A, the pattern from the new ring (fig. 2(c)) is seen to correspond to α-iron and graphite, predominately α-iron. The pattern plotted in figure 2(a) (same as fig. 1(f)) is from a specimen of ring 1, test 1, and has previously been identified as α-Fe_2O_3 and Fe_3O_4. Three other specimens from different parts of the same piston ring showed the presence of oxides, although the specimen from which the pattern of figure 2(a) was obtained showed the greatest quantities of oxides, relative to graphite and α-iron. On the other hand, patterns from ring 2 of the same test (figs. 2(b) and 6(b) are representative) showed graphite with smaller amounts of α-iron and only faint traces of oxides; specimens from the third compression ring and the oil rings (test 1) showed graphite with relatively small amounts of α-iron but no oxide diffraction rings.

The first and second compression rings from two other tests on the same engine (tests 2 and 3) were examined. Strong oxide patterns were obtained from specimens of ring 1, test 3; patterns from ring 2 were difficult to measure because of the high background. Oxides occurred only in small quantities (relative to graphite and α-iron) on the top ring of test 2, possibly because the ring was pitted, which indicated poor performance. The second ring of this test showed no oxides.

Used rings from test 4 were examined but gave diffuse patterns (fig. 6(c)) and a high background because of the highly polished surfaces of these rings. In order to obtain information on the oxides present, the rings were very mildly abraded using different grades of emery paper. The method consisted in making two to four strokes in the same direction along the specimen, using a minimum of pressure. The specimen was then placed in the diffraction chamber without washing off the debris. This "plowing" operation so raised the surface material that sharp diffraction rings could be obtained. Some iron located below the surface was torn up in the process and contributed to the diffraction pattern. From each ring examined by this abrasion method, two or three specimens were taken and several exposures made for each, first without any surface distrubance and then with increasing amounts of abrasion.

The results of this method applied to piston rings of test 4 are plotted in figure 3. Figures 3(a) and 3(b) show two patterns obtained from the same specimen of ring 1; each pattern has four extra rings (other than graphite and α-iron rings) of which two have the same d values in both patterns. The extra rings in the pattern of figure 3(a) were attributed to α-Fe_2O_3 because, together with two rings common to both graphite and α-Fe_2O_3, they account for six of the strong α-Fe_2O_3 rings. In the case of the pattern of figure 3(b) (photograph shown in fig. 6(d)), three of the four extra rings correspond to Fe_3O_4 (the fourth is unidentified), which indicates the presence of that substance. Examination of several specimens of each of the other compression rings from test 4 showed that the extra diffraction rings are weaker for the second piston ring and very faint for the third. For example, figure 3(c) shows one of the patterns obtained from the third piston ring using the same abrasion technique. The three extra rings agree with those appearing in figure 3(b) but are extremely faint. The oxide gradation, therefore, is similar on the rings of test 4 to that observed on the rings from the other three tests.

The method of abrasion served another purpose: It indicated the absence of any layer of crystalline material other than oxides

and graphite on the surfaces of the used piston rings examined. The disadvantage of the method is in the disruption of any preferred orientation in the surface materials.

Good agreement is shown in the comparison of the results obtained with these piston rings with the results of the tests of the piston-ring assembly reported in reference 1. In the previous work, the top ring of the assembly was chromo-plated and the only substance found on the used cast-iron rings was graphite, except for one oxide diffraction ring that appeared occasionally and with varying intensity. In the present experiments, the appearance of strong oxide patterns from only the top cast-iron ring is consistent with the previous failure to observe such patterns. The surfaces of the used rings from test 1 of this report were rougher than those from the tests of reference 1; therefore, electrons were diffracted by the iron underlying the surface, which explains the appearance of α-iron in the patterns from all of the rings of test 1 except the top ring. Preferred orientation did not appear in the graphite patterns in the present experiments because the roughness of the surface in the specimens used prevented the occurrence of any high degree of preferred orientation of the graphite crystals.

Results from the Cast-Iron Cylinder

The specimens from the cast-iron cylinder examined and the identification of patterns obtained from them are listed in table 2. In the previous discussion of identification of the oxides, it was noted that the pattern from specimen 3 indicated the presence of Fe_3O_4 and possible traces of α-Fe_2O_3. Specimen 1 from the region of the cylinder above the piston-ring stroke was covered by oil combustion products most of which were removed by washing with several organic solvents before diffraction photographs were taken. Some "lacquer" remaining on the surface probably accounts for the high background and failure of sharp rings to appear until after abrasion, when a strong pattern of Fe_3O_4 appeared. Two specimens, 8 and 9, from the region of the cylinder below ring travel failed to show the presence of iron oxides in detectable quantities.

The criterion used in table 2 to determine the quantity of oxides relative to graphite and α-iron was the comparison of the relative intensities of the strongest oxide diffraction ring at 2.5 to 2.6 A with the strongest diffraction ring of both α-iron and graphite at 2.03 A. Only qualitative information can be obtained in this manner. It is clear from table 2 and figures 4(a) to 4(d) that there is a general increase in the relative quantity of oxide from the bottom to the top of the cylinder.

EVALUATION OF RESULTS

Comparison of the results obtained from the cylinder with the results from the aircraft piston rings shows that there is good agreement both in the type and occurrence of oxides appearing. In both cases the oxides were identified as α-Fe_2O_3 and Fe_3O_4, although the cylinder specimens showed mainly Fe_3O_4. In addition, it has been shown that the quantities of oxides appearing both on the piston rings and on the cylinder are greatest for the regions nearest the cylinder head. Several engine-operation variables such as temperature, pressure between piston-ring and cylinder surfaces, and exposure of the surfaces to corrosive agents in the combustion products vary with position in the same way as the observed oxide quantities. It therefore should be possible to correlate oxide formation with one or more of these variables. The authors of reference 5 reported that α-Fe_2O_3 and Fe_3O_4 could be formed on cast iron by rubbing it against other materials. The occurrence, however, of Fe_3O_4 on cylinder specimen 1, above the piston stroke where no rubbing had taken place, shows that pressure between the surfaces is not the only variable which affected the oxide formation.

The relation between oxidation and wear has been studied by several investigators. A summary of work on wear oxidation is included in a paper by Donaldson (reference 6). X-ray diffraction and chemical analysis were applied in these investigations to the wear particles removed from the surfaces. When wear had proceeded in an oxygen-containing atmosphere, the particles were found to be partly oxidized. There are two distinct differences in the study of wear oxidation as compared with the examination made in this report of oxides on piston rings from aircraft engines. First, wear particles have been removed from the surface and their examination does not directly reveal the nature of the remaining surface. Second, as previously discussed, the formation of oxides on the surfaces discussed in this report differs from the oxidation of wear particles observed in a wear machine in that it depends upon other variables in addition to the pressure between the rubbing surfaces. Wear studies can reveal only indirect information in determining the nature of a run-in surface. On the other hand, the observation, through electron diffraction, of a gradation of oxides which was not present before engine operation indicates that iron oxides must be considered in studying the formation of run-in surfaces on cast-iron piston rings.

SUMMARY OF RESULTS

Electron-diffraction examination of cast-iron piston rings from tests on liquid- and air-cooled single-cylinder engines and of specimens from a cast-iron cylinder that had been operated for a long time showed the following results:

1. In addition to diffraction rings of α-iron and graphite, extra rings appeared from many of the surfaces. These diffraction rings belong to oxides of iron. From the patterns that yielded the most prominent oxide rings, identification of α-Fe_2O_3 and the indistinguishable pair, Fe_3O_4 and γ-Fe_2O_3, was made. Three additional diffraction rings that indicated preferred orientation of the corresponding crystallographic planes were not identified.

2. Iron oxides were found to an appreciable extent only on the top compression ring from three tests on the liquid-cooled engine. The other piston rings of the assembly generally showed the presence of a graphite layer. A new compression ring of this type showed only α-iron with smaller quantities of graphite.

3. Piston rings from the tests on the air-cooled engine gave a diffuse pattern because of the high polish of the ring surfaces. Diffraction examination after very mild abrasion, without removal of the abrasion debris, showed that oxides were present in decreasing quantities (relative to graphite and α-iron) on the top, the second, and the third piston rings of this assembly. The third ring showed only a faint trace of oxides.

4. Nine specimens from the cast-iron cylinder showed continuously increasing amounts of oxides relative to graphite and α-iron as the top of the piston stroke is approached. The specimens from positions below the region of ring travel showed no detectable quantities of oxides, but oxides were present above the piston stroke where no rubbing had taken place.

CONCLUSIONS

Layers of iron oxides cover the wearing surfaces of cast-iron engine parts, such as piston rings and cylinder barrels, in those regions where operating conditions of temperature, pressure on the surfaces, and exposure to corrosive agents in the combustion products are extreme. The quantities of oxides decrease over the regions where these conditions are milder.

Aircraft Engine Research Laboratory,
    National Advisory Committee for Aeronautics,
        Cleveland, Ohio,

REFERENCES

1. Nowick, A. S., and Brockway, L. O.: An Electron-Diffraction Examination of Cast-Iron Piston Rings from Single-Cylinder Aircraft-Engine Tests. NACA ACR No. E4J25, 1945.

2. Finch, G. I., Quarrell, A. G., and Wilman, H.: Electron Diffraction and Surface Structure. The Structure of Metallic Coatings, Films, and Surfaces. Trans. Faraday Soc. (London), vol. XXXI, 1935, pp. 1051-1080.

3. Germer, L. H.: Diffuse Rings Produced by Electron Scattering. Phys. Rev., vol. 49, no. 2, 2d ser., Jan. 15, 1936, pp. 163-166.

4. Krause, Alfons, and Lewandowski, A.: Amorphe und kristallisierte Oxyhydrate und Oxyde. XLVIII. Die Aktivität des Eisenrostes. Z. anorg. allg. Chem., Bd. 239, Heft 4, Nov. 8, 1938, pp. 395-398.

5. Iitaka, Ichiro, and Tokumitu, Tadasi: Study on Wear with Electron Diffraction Method. Trans. Soc. Mech. Eng. Japan, vol. 6, no. 25, Nov. 1940, pp. 124-127. (Japanese text with English summary, pp. S3-S4.)

6. Donaldson, J. W.: Metallic Wear of Metals and Alloys. Metallurgia (British), vol. XXVI, no. 155, Sept. 1942, pp. 155-160.

TABLE 1

TEST CONDITIONS FOR THE USED CAST-IRON PISTON RINGS EXAMINED

Test (a)	Piston rings examined	[b]Break-in time (hr)	Time of operation at test conditions (hr)	Engine speed at final test conditions (rpm)	bmep at final test conditions (lb/sq in.)	Ring weight loss	Remarks on ring condition
1	All	$6\frac{1}{2}$	5	3000	290	Normal	
2	1st and 2d	$8\frac{5}{12}$	$24\frac{5}{6}$	3000	151-267	Slightly high	Top ring slightly pitted
3	---do---	$7\frac{1}{4}$	24	2600	159-228	Normal	
4	1st, 2d, and 3d	$5\frac{5}{6}$	10	2500	250	---do---	

[a]Tests 1, 2, and 3 were run on a single-cylinder liquid-cooled
engine; test 4 was run on a single-cylinder air-cooled engine.

[b]"Break-in" rather than "run-in" is used here to denote a procedure
of preliminary engine operation in which load and speed are
increased to normal operating values in order to avoid confusion
with the term "run-in surface" as defined in this report.

National Advisory Committee
for Aeronautics

TABLE 2

RESULTS OF DIFFRACTION EXAMINATION OF SPECIMENS FROM THE

CAST-IRON CYLINDER OPERATED FOR A LONG PERIOD OF TIME

Specimen	Distance from top of cylinder (in.)	Region of cylinder	Diffraction identification of surface material
1	1/8	Above ring travel	[a]Very strong oxide pattern
2	1/2	Ring travel	[a]Strong oxide
3	1	---do------	Very strong oxide; trace of graphite
4	$1\frac{3}{8}$	---do------	Strong oxide; smaller quantity of graphite
5	$1\frac{5}{8}$	---do------	Mostly graphite; strong oxide
6	$4\frac{3}{4}$	---do------	Mostly α-iron; medium oxide; faint graphite
7	$5\frac{3}{8}$	Opposite rings at B.D.C.	Graphite; α-iron; faint oxide
8	$5\frac{7}{8}$	Below ring travel	Graphite; α-iron
9	$8\frac{3}{8}$	---do------	Do.

[a]These specimens required abrasion before patterns of sharp diffraction rings were obtained; therefore, no comparison of the relative amounts of oxides and graphite could be made.
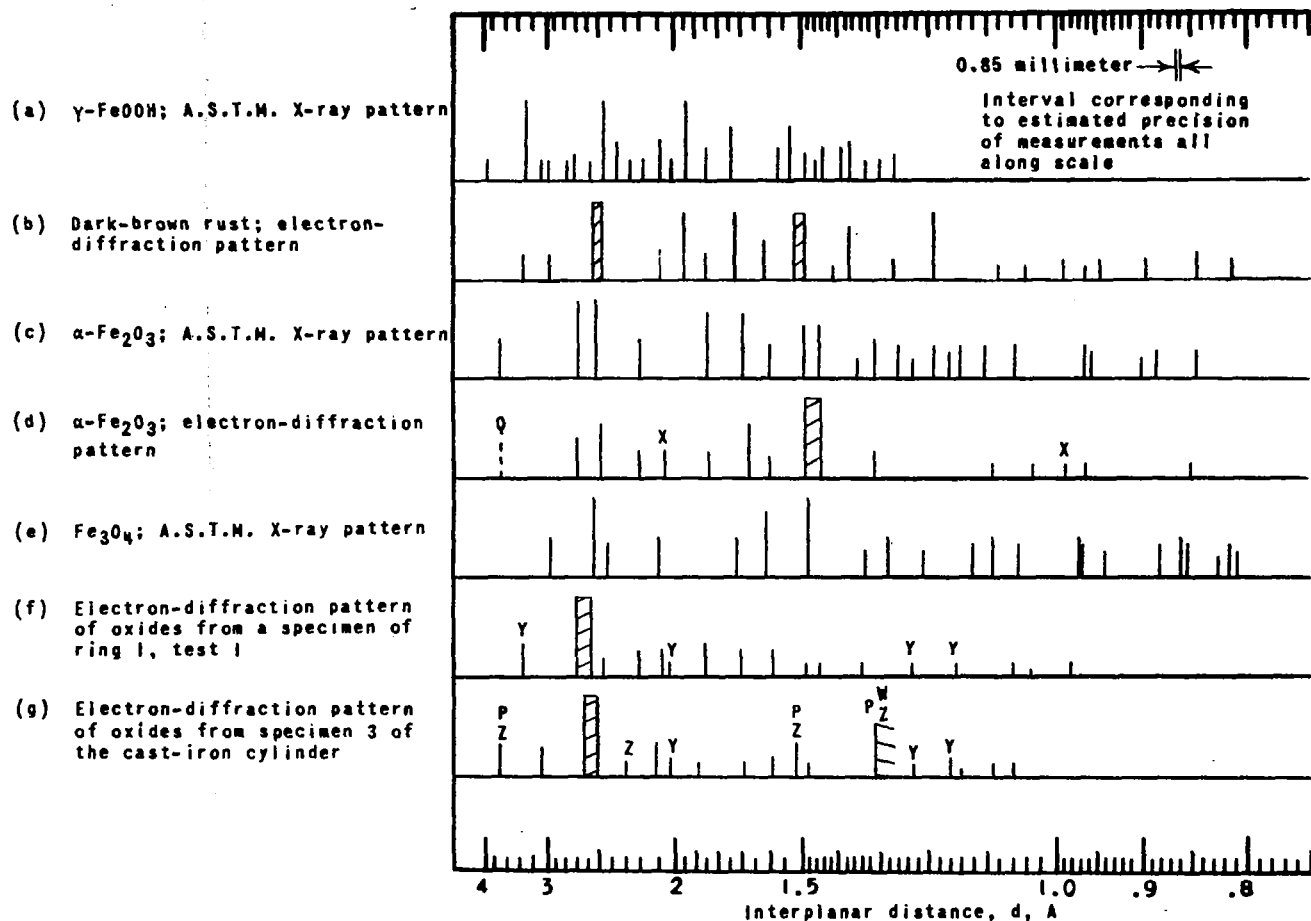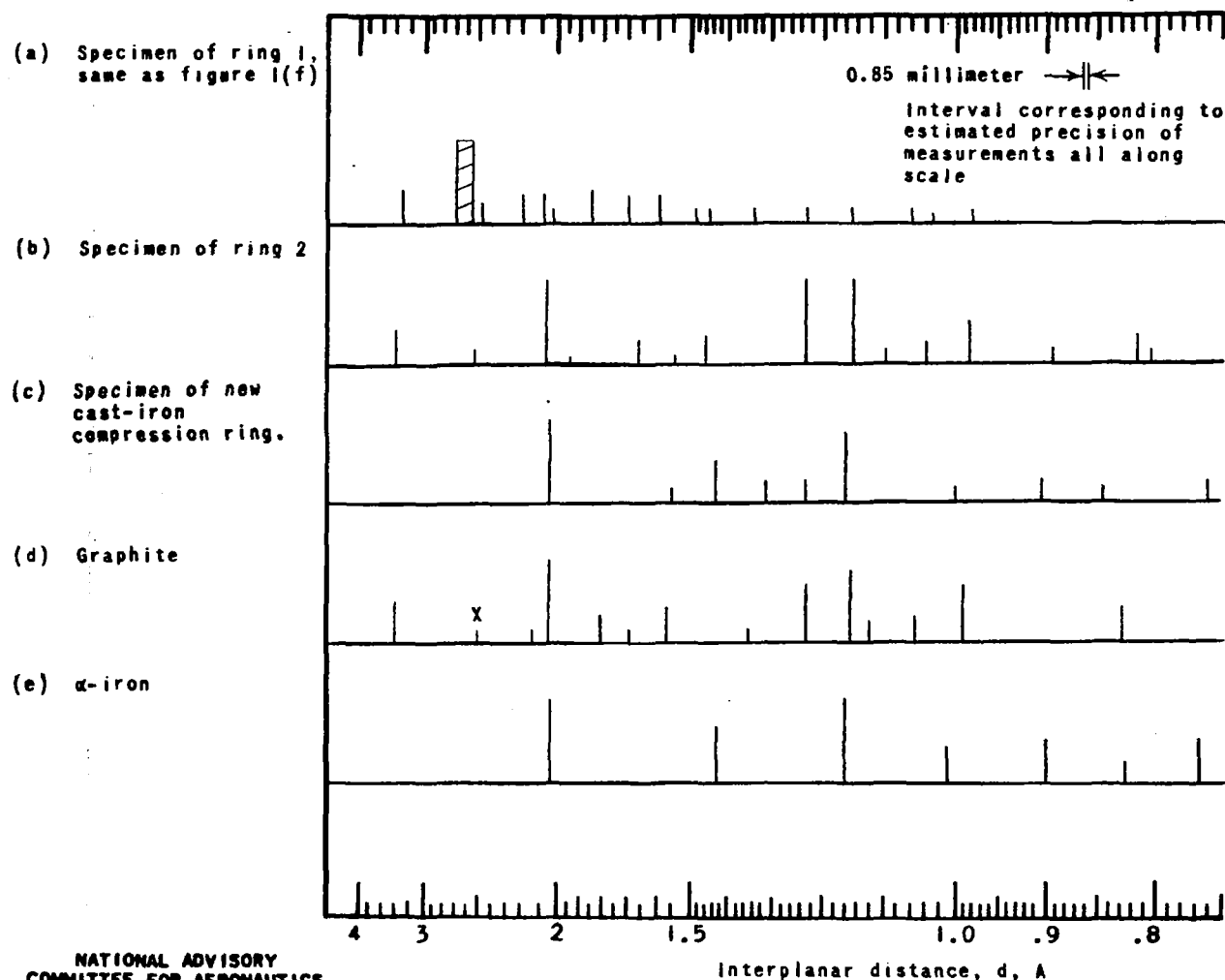
NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS

0.85 millimeter →||←

Interval corresponding
to estimated precision
of measurements all
along scale

(a) γ-FeOOH; A.S.T.M. X-ray pattern

(b) Dark-brown rust; electron-
     diffraction pattern

(c) α-Fe₂O₃; A.S.T.M. X-ray pattern

(d) α-Fe₂O₃; electron-diffraction
     pattern

(e) Fe₃O₄; A.S.T.M. X-ray pattern

(f) Electron-diffraction pattern
     of oxides from a specimen of
     ring 1, test 1

(g) Electron-diffraction pattern
     of oxides from specimen 3 of
     the cast-iron cylinder

Interplanar distance, d, A

Figure 1. - Comparison of iron-oxide patterns. The notation within the scale is as
   follows:

   P   These lines show preferred orientation.
   Q   This α-Fe₂O₃ line does not appear in the pattern because of halation of the image of the central beam.
   W   The intensity decrease at the outer edge of this ring is gradual.
   X   These lines are not from α-Fe₂O₃ and probably are the results of impurities.
   Y   These lines are probably graphite lines.
   Z   Unidentified lines.

NATIONAL ADVISORY
COMMITTEE FOR AERONAUTICS

Fig. 1

(a) Specimen of ring 1,
same as figure 1(f)

0.85 millimeter →|←

interval corresponding to
estimated precision of
measurements all along
scale

(b) Specimen of ring 2

(c) Specimen of new
cast-iron
compression ring.

(d) Graphite

X

(e) α-iron

4   3   2   1.5   1.0   .9   .8

Interplanar distance, d, A

Figure 2. - Electron-diffraction patterns from piston rings run in a liquid-cooled
single-cylinder engine (test 1, table 1) and electron diffraction comparison patterns.
The notation is explained as follows:

X This line is not from graphite and probably is the result of oxide impurities.

Fig. 2
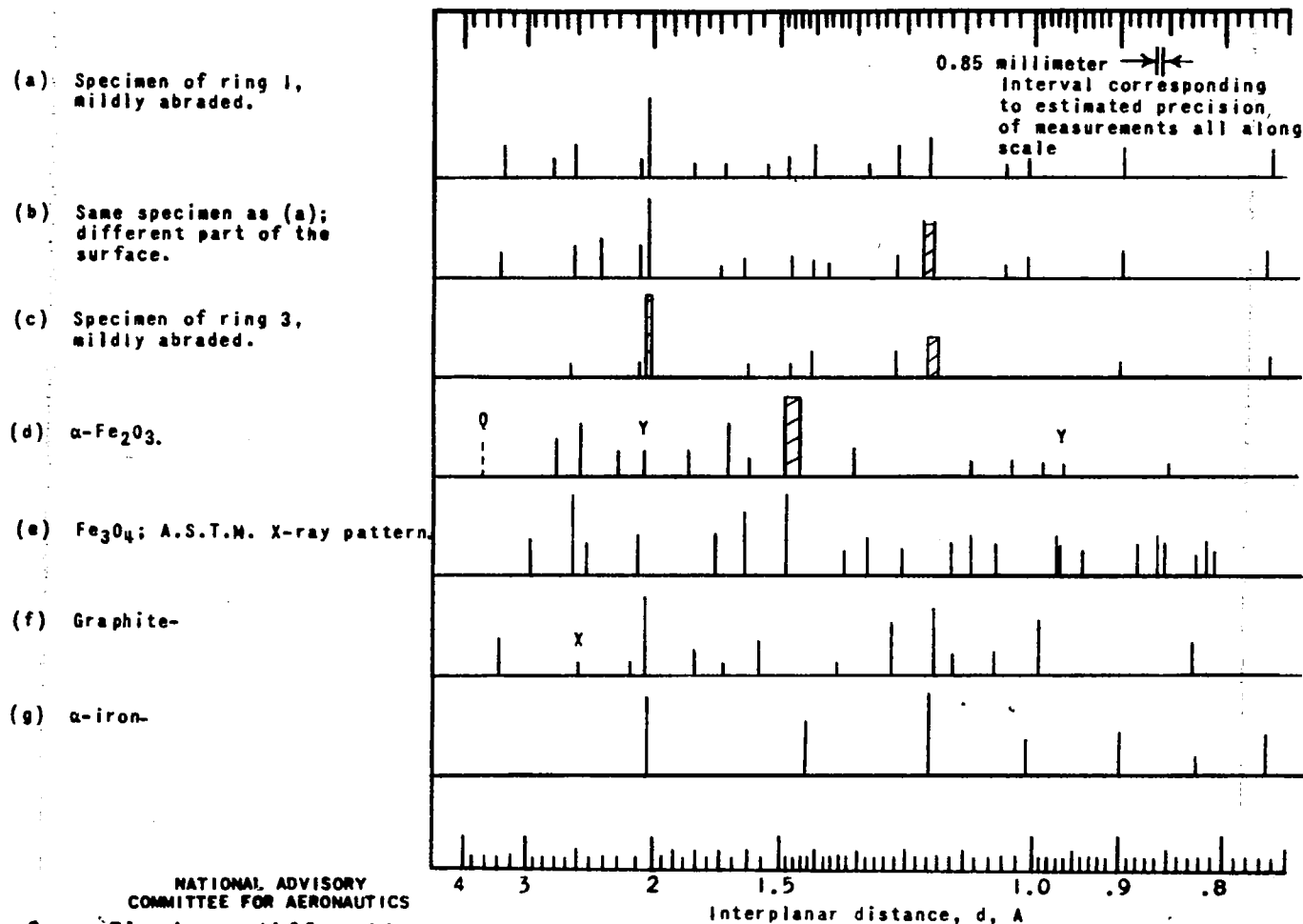
Figure 3. - Electron-diffraction patterns from piston rings run in an air-cooled single-cylinder engine (test 4, table I) and electron-diffraction comparison patterns. The notation is explained as follows:

Q   This α-Fe₂O₃ line does not appear in the pattern because of halation of the image of the central beam.

X   This line is not from graphite and probably is the result of oxide impurities.

Y   These lines are not from α-Fe₂O₃ and probably result from impurities.

Fig. 3
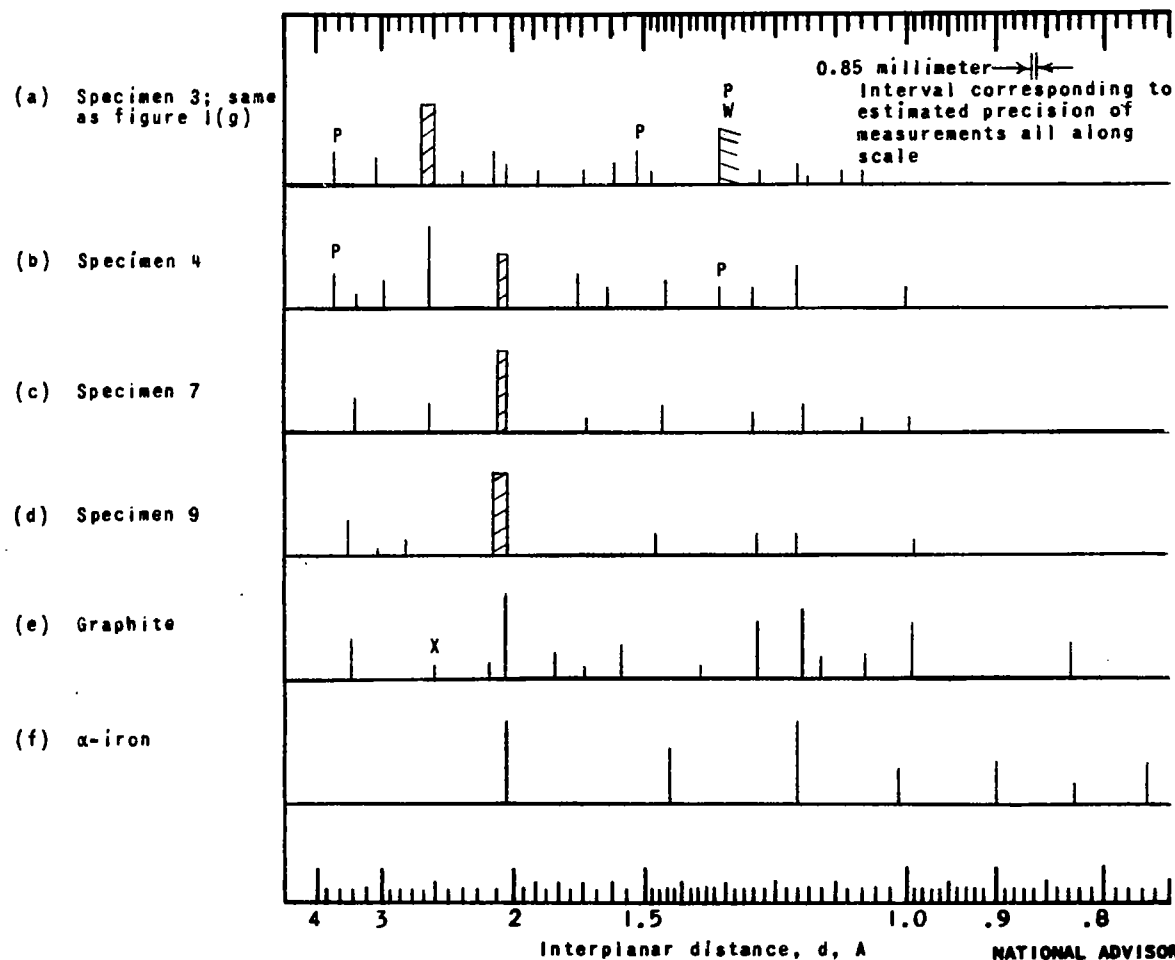
Figure 4. - Electron-diffraction patterns from specimens of a cast-iron cylinder operated for a long period of time (table 2) and from graphite and α-iron. The notation is explained as follows:

P   These lines show preferred orientation.
W   The intensity decrease at the outer edge of this ring is gradual.
X   This line is not from graphite and probably is the result of oxide impurities.
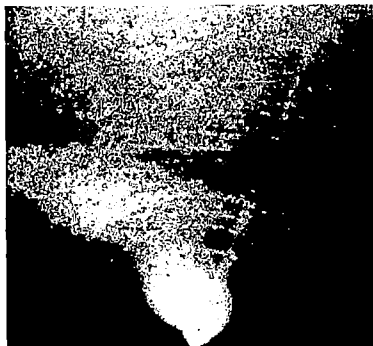
Fig. 4
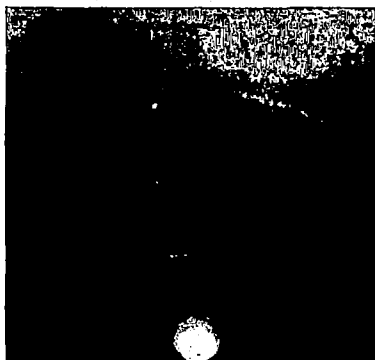
(a) α-iron.
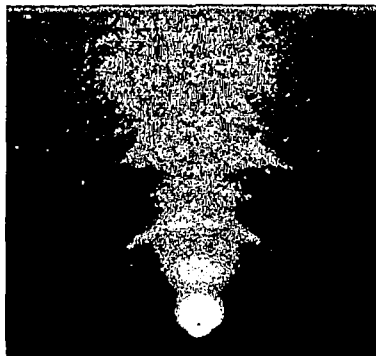
(b) Graphite (from cast iron etched with 2 percent nital).

NACA
C-9921
5-9-45
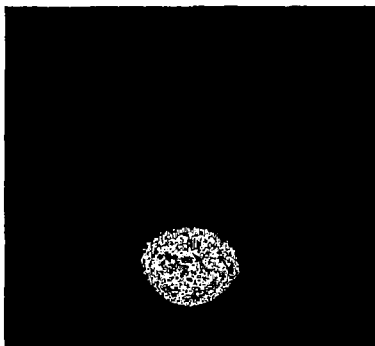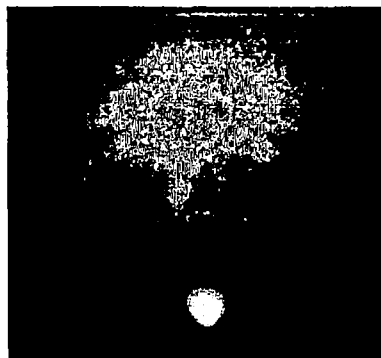

(c) α-Fe₂O₃ (from a steel specimen etched, then heated).

(d) γ-FeOOH with small amounts of Fe₃O₄ (from rusted cast iron).
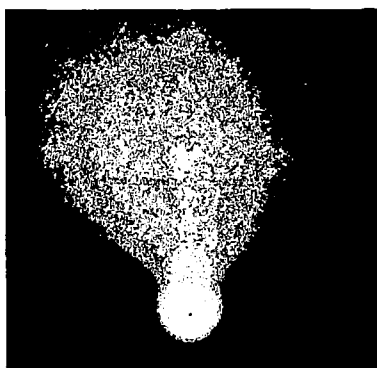
FIGURE 5.—Comparison electron-diffraction patterns. X2.
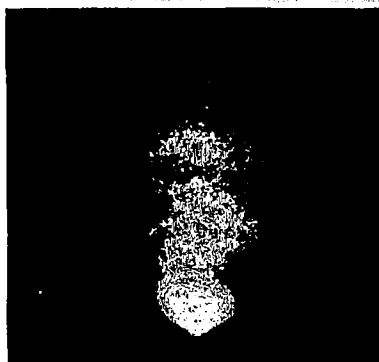
(a) Pattern from cast-iron ring 1, test 1, identified as α-Fe_2O_3 and Fe_3O_4.
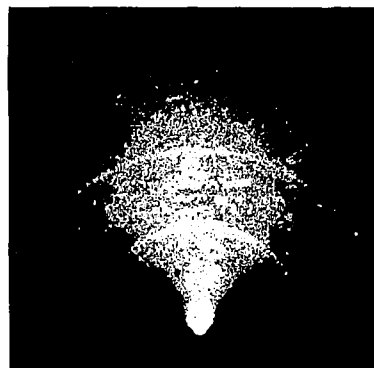

(b) Graphite pattern from cast-iron ring 2, test 1.

NACA
C-9922
5-9-45

(c) Diffuse pattern from ring 1, test 4.

(d) Pattern from same specimen as 6(c) (after mild abrasion) showing oxide rings in addition to α-iron and graphite.

FIGURE 6.—Electron-diffraction patterns from cast-iron piston rings. X2.